SRI CAT NEWSLETTER

Synchrotron Radiation Instrumentation Collaborative Access Team Newsletter

Vol. 1, No. 4 **April 1995**

From the desk of the Program Director:

What we've been waiting for for a long time is very close now! SRI CAT is only few days away from getting the very first APS x-ray beam. A lot of thorough planning, design and extensive construction precede this event.

In the last ten days of March, SRI CAT began the beamline commissioning process. Prior to that, SAD Review and Commissioning Readiness Review took place. On March 22, the Safety Envelope and the initiation of commissioning activities for the APS beamlines was approved by DOE.

By that time, the installation of the 1-BM-A FOE was completed and both processes: the required documentation preparation and completion of the construction and tests merged in the long awaited commissioning phase. The SRI CAT commissioning team lead by D.Mills developed the procedure for the "first x-ray" beam experiment. The goal of the experiment was to allow the synchrotron radiation into 1-BM-A for a short period of time to conduct the first measurements of the x-ray beam.

In Sector 1, the bending-magnet front end has been fully installed and successfully tested by extracting the first radiation on March 26. On the experimental floor, all white-beam stations are near completion. In particular, there has been a great effort to complete the 1-BM-A station by the end of March, so it can be ready for the first synchrotron validation of the shielding by mid-April. The status of the optical components is as follows: modifications on the Kohzu monochromator for the insertion-device beamline are being made, so the cooling of the first crystal will be compatible with liquid nitrogen, and the contract for the monochromator on the bending-magnet beamline was awarded to Physical Sciences Laboratory, University of Wisconsin. The delivery is scheduled for September of 1995. The vendor for the collimating mirror was chosen, and the contract is expected to be awarded by mid-April.

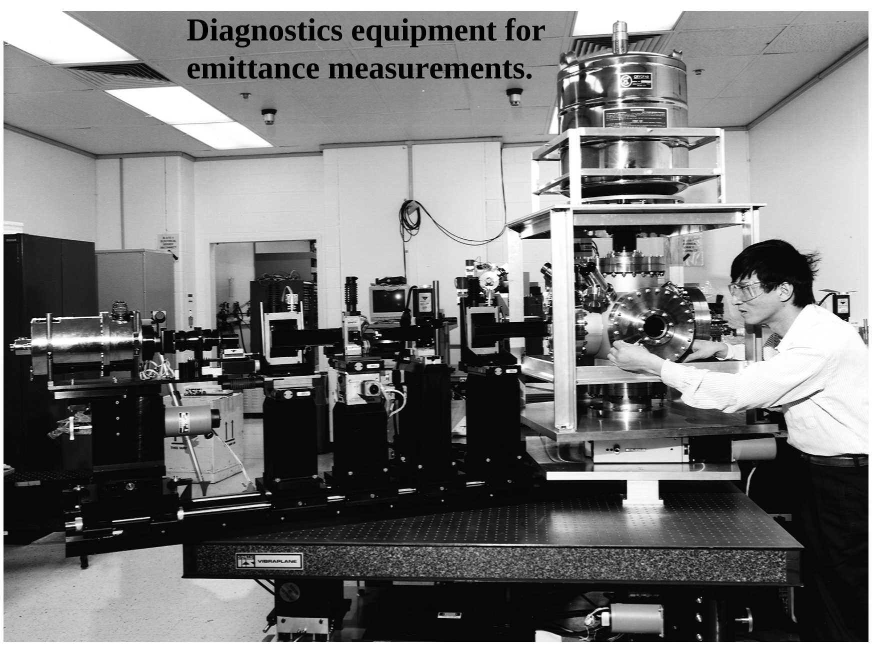
The most critical optical component for Sector 2, a 1.2-meter-long, high-heat-load mirror, is being fabricated at Rockwell International in Albuquerque. The vacuum and mechanical systems for the mirror have been designed at the APS and are in procurement now. The expected date for the mirror delivery is May. Also, several side-cooled mirrors for both undulator and bending-magnet beamlines have been designed and are in procurement.

Considerable effort has gone into the design of the SRI-CAT beamline to insure that proper radiation safety is provided. In particular, the 1-ID and 2-ID beamlines have been used by the APS/XFD Radiation Safety Committee as case studies for bremsstrahlung, white-beam, monochromatic-beam and pink-beam shielding and for the ray tracing. General results from these studies have been published in ANL/APS/TB-7 and ANL/APS/TB-20, while specific calculations and analyses for the 2-ID beamline are given in ANL/APS/TB-21. In particular, new analyses have been done on the generation of secondary bremsstrahlung from grazing-incidence targets (i.e., mirrors) and collimators.

The high-heat-load monochromator for Sector 3 was delivered to SRI CAT in March.

The construction of 3-ID-A and -B started on March 21, 1995, and will be completed by May. The white-beam slits (L5-20) have been ordered, as well as the P5 integral shutter. We expect the delivery of an undulator with a 2.7-cm period in June 1995, and

Diagnostics equipment for emittance measurements.



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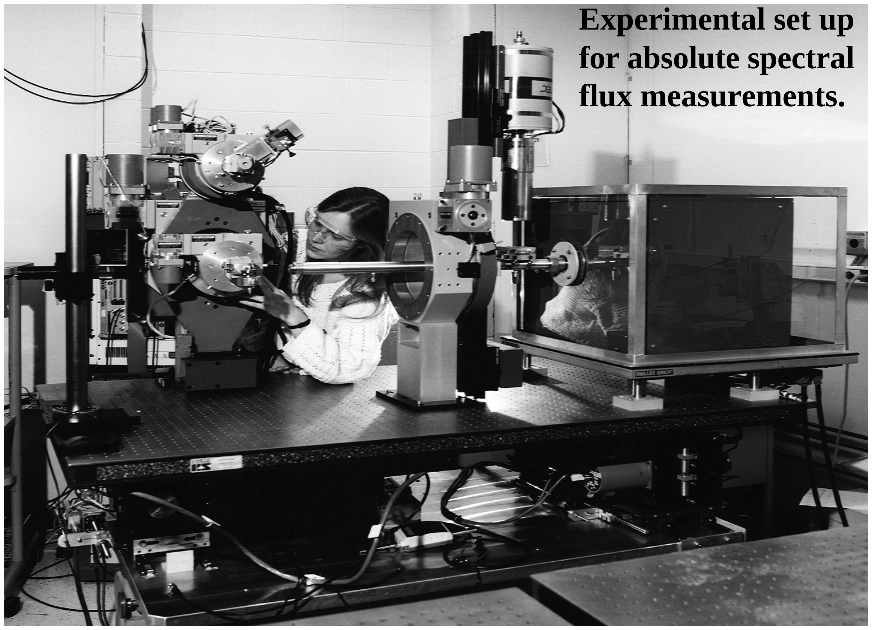
the rest of the components by August 1995. Finally, we signed a contract with Zeiss for an 80-cm-long, double-figure mirror in March 1995. The expected delivery is by summer of 1996.

In a preparation to receive the first x-ray beam from the 1-BM bending magnet and later on from the first ID, SRI CAT members designed and constructed a suite of diagnostic instruments. These instruments will be used to measure the absolute value of the spectral photon flux and the positron beam emittance, from which the brilliance can be inferred. You can find a detailed description of these instruments in this current issue. *E. Gluskin*¶



Experimental set up for absolute spectral flux measurements.

X-Ray Beam Diagnostics at the APS

As the Advanced Photon Source begins operation, beam diagnostics will be an essential part of the commissioning process to determine parameters of the stored positron beam and the performance of the bending-magnet (BM) and insertion-device (ID) sources. The results will serve both machine control and users. Among the first diagnostics experiments to be performed will be source size measurements on the 1-BM beamline, and phase-space, absolute spectral flux, and polarization measurements on the 1-ID beamline.

1. Source Size Measurements

Because of the low emittance of the positron beam in the storage ring, the positron beam divergence is overwhelmed by the natural opening angle of the BM radiation, making the divergence difficult to determine. Instead, the photon beam size is a true characterization of the positron beam size. Therefore, only source size measurements will be performed at BM beamlines.

At the outset, we will image the x-ray source with a pinhole and measure the image dimensions to determine the source size. The experimental setup is shown in Fig. 1. This method, which does not require a monochromator, offers ease of alignment and short exposure times, even at low ring current. The pinhole consists of a pair of crossed Huber slits with 10 µm apertures. The x-ray image is converted to visible light by a $CdWO_4$ scintillation crystal, which is then magnified seven times by a lens system and detected by a low noise CCD camera. The magnified source image can be directly read out from the CCD. The x-rays are converted to visible light to increase the detector dynamic range and to improve the spatial resolution by optical magnification.

Later, we plan to utilize a zone plate lens instead of a pinhole because of its higher numerical aperture (6×10^{-4}) and efficiency (30%). A monochromator is necessary for this approach (Fig. 2), in which the zone plate images the source at ~8 keV with a magnification of 1/25. An order sorting aperture (OSA) improves the image contrast by selecting the first-order diffracted x-rays. The expected image size of the APS BM source is about 17 µm (4σ).

We will use two techniques to determine the image dimensions. First, the image profile will be measured as a function of the position of a sharp knife edge which is scanned across the focal plane. The transmission profile so obtained contains all information necessary to reconstruct the source image profile. We will use a lithographically fabricated gold knife edge with a width less than 0.2 µm and a mechanical translation system with a resolution of 0.05 µm. This technique, recently tested by us at a BM beamline at the NSLS, gave results that agreed to within 3-8% of the expected source size. The second technique is to enlarge the source image,

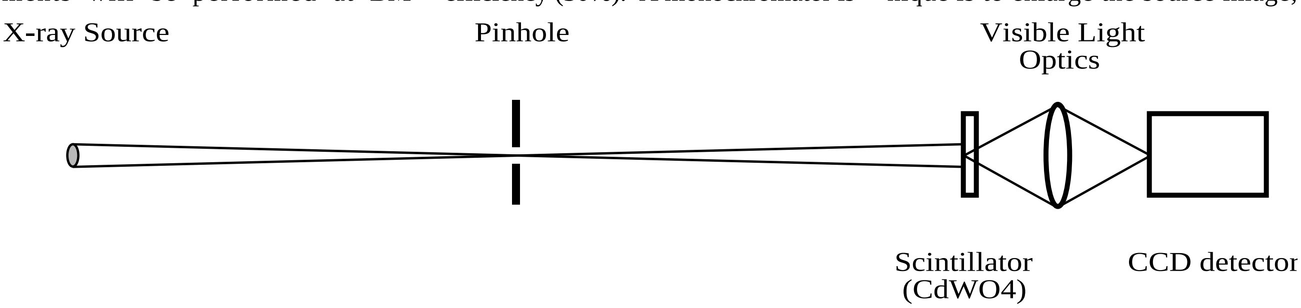


Figure 1 Schematic of the source-size measurement experiment using pinhole optics.

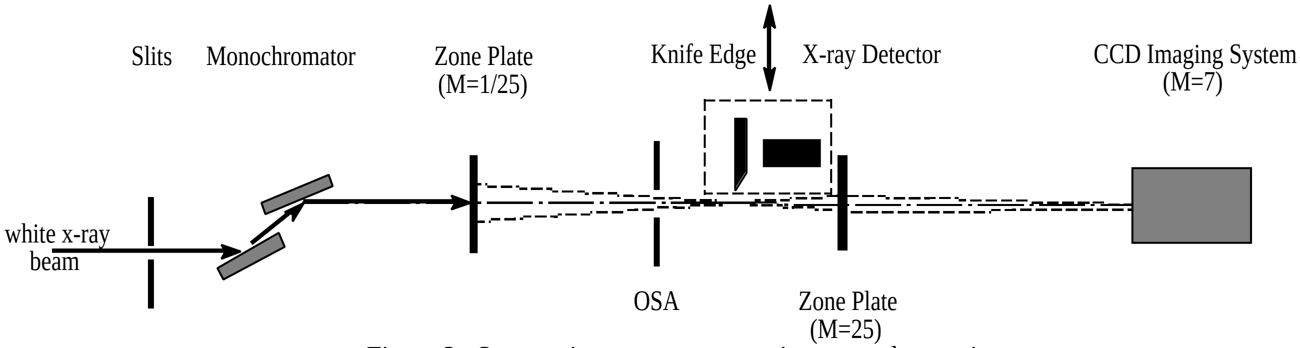


Figure 2. Source-size measurement using zone plate optics.

with the help of a short-focal-length zone plate, to a dimension measurable by the CCD imaging system. With the magnification provided by the two zone plates and the detector optics, the photon source will be magnified by seven times and the image will cover 150x150 pixels in the CCD.

The accuracy of the source size measurement depends on the accuracy of the optical alignment and the spatial resolution of the zone plates and CCD imaging system.

2. Phase-Space Measurements

The goal of these measurements is to map the phase space of the source, which determines all properties of the beam. Under the Gaussian beam approximation, only the source size and divergence are necessary to characterize the beam at any point. A contour plot of the phase space for $e^{-1/2}$ of its on-axis intensity yields an ellipse that characterizes the emittance of the beam, a fundamental property of the storage ring.

Because the radiation characteristics of an undulator depend sensitively on the parameters of the stored positron beam, phase-space measurements of the photon source can give the size and divergence of the positron beam. Once the source size and divergence of the photon beam are measured, the size and divergence of the positron beam are obtained by deconvolving them it from the zero-emittance photon source size $(\lambda L)^{1/2}/4\pi$ and divergence $(\lambda/2L)^{1/2}$, where λ is the wavelength of radiation, and L is the length of undulator. When deconvolving the horizontal phase space, one also has to consider the momentum dispersion of the positron beam to account for the correlation between the position of a positron and its momentum fluctuation.

Fig. 3 illustrates the experimental arrangement for the phase-space measurement. A monochromatic x-ray beam (8-12 keV) is selected from the undulator source with a high-heat-load x-ray monochromator. The monochromatic beam is incident upon a slit array consisting of one row and one column of slits. The x-ray image transmitted by the slit array is detected with the CCD system. Both the vertical and horizontal phase-space information can be obtained from a single exposure.

The widths of the measured slit images contain the information about the source size, and the distances between the slit images and the optical axis contain the information about the source divergence. From the intensity profiles of the slit images, we will construct the phase-space ellipse using the intensity scaling of Mills et al.[1] Because the intensity scaling is based on an ideal geometrical transformation, effects due to diffraction and the finite slit size need to be included. This technique, tested by us on an ID beamline at CHESS, gave results that agreed within 5-15% of the expected source size and divergence.[2]

The accuracy of the measurement depends on the accuracy and uniformity of the slits, the location of the slit array, the spatial resolution of the x-ray imaging system, and the performance of the monochromator. We have had arrays of slits 25 µm by 45 µm fabricated lithographically to 0.2-µm accuracy. The spatial resolution of the x-ray imaging system is better than 3 µm.

3. Absolute Spectral Flux and Polarization Measurements

The APS undulator A will provide high brilliance x-ray radiation in the 3.2-45 keV spectral energy range. In order to characterize the performance of the undulator, the absolute value of the flux through a pinhole and the degree of polarization as a function of angle and energy will be measured. From the absolute flux, the spectral intensity and brilliance will be determined. These measurements will have to be made under conditions of high incident total power and power density. The power density produced by undulator A at 30 m will be 130 W/mm^2, with a total incident power of 2.1 kW through a 5 mm by 5 mm aperture. To investigate the

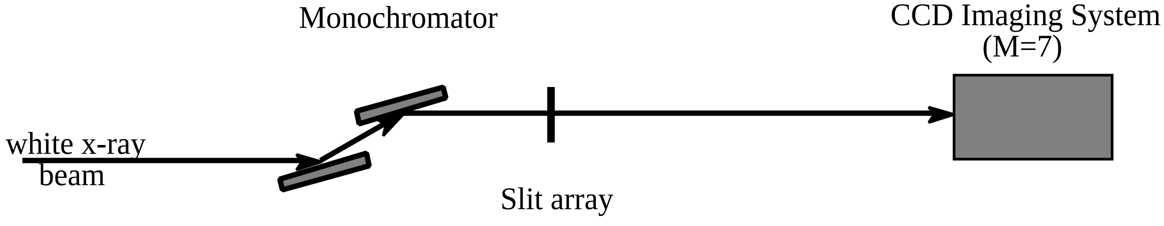


Figure 3. Schematic of the emittance measurement experiment.

spatial distribution of the undulator beam, the combination of a water-cooled pinhole and water-cooled slits will be used, which will sufficiently decrease the power throughput. One way to deal with the high power density that remains is to use the radiation scattered by a volume of gas. This method was tested during a 1993 CHESS undulator run.[3]

To make absolute spectral flux measurements at the APS, we will use this scattering technique and an energy-dispersive solid-state Si(Li) detector. Scattering by a gas will reduce the incident intensity for the limited counting rate of the Si(Li) detector. The detector counting rate is defined by the scattering geometry, gas density, differential cross section, and detector efficiency. The gas density is defined by monitoring the pressure and temperature. The detector efficiency is calculated, based on the absorption of x-rays in silicon. This method showed good (within 10%) agreement with an experimental calibration of a similar Si(Li) detector.[4]

The measured scattering spectrum is a sum of the coherent and incoherent scattering spectra. It is possible to recover the primary spectrum from the measured data by unfolding the Compton profile.[5] To simplify this procedure, it is best to choose a low-Z material for the target. The reasons for this are, (1) the coherent contribution decreases with Z, (2) the mean kinetic energy of the scattering electrons in the target decreases with Z, thus decreasing Compton broadening, and, (3) the Compton process will dominate at large scattering angles, even at low energies. However, Compton broadening increases with increasing scattering angle. Consequently we will use helium gas and a scattering angle of 90 degrees. The atomic form factor and the incoherent scattering function for helium have been tabulated with high accuracy.[6]

The experimental setup (Fig. 4) is mounted on a standard optical table. To analyze the spatial distribution of the radiation, the apparatus must be scanned about the beam axis in the plane perpendicular to the beam. The water-cooled pinhole and slits are lead shielded and are placed into the helium filled container, which is connected directly to the beryllium window of the beamline front end. The Si(Li) detector is mounted on a goniometer and can be rotated around the beam axis in order to define the linear polarization components of the incident radiation. The volume of the helium cell and the detector solid angle may be changed to optimize the detector counting rate.

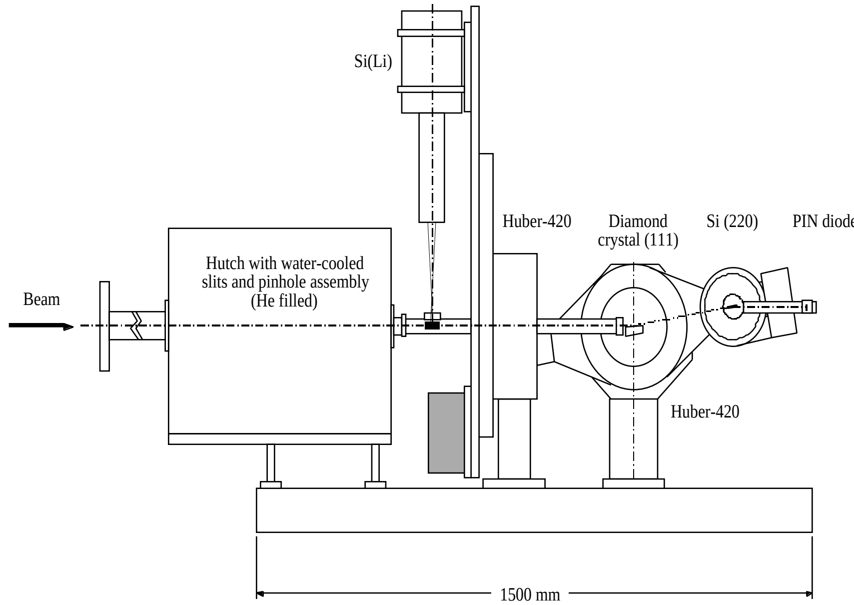


Figure 4. Schematic of the stepup used to measure absolute spectral flux and polarization in an undulator beamline.

To take spectral flux measurements with better resolution, which may be necessary to investigate the odd undulator harmonics, a diamond-crystal monochromator will be used. However, absolute measurements made by this method involve more parameters and may not be as accurate as those made by the scattering method. In our experiment, the diamond-crystal monochromator (Fig. 4) is located behind the scattering setup. It consists of a 4-circle Huber goniometer and a 1-mm-thick diamond (111) crystal mounted in the Bragg geometry. The crystal is cooled by a water jet. To analyze the rocking curve of the diamond, a Si (220) crystal will be used. The detector is a 0.5-mm-thick Hamamatsu PIN diode working in the current regime.

EPICS software is used for the data acquisition and control system, which was previously tested at NSLS. We found the system to be a very flexible experimental tool and will provide a starting point for our beamline control systems.

Construction of the instrumentation for beam size, absolute spectral flux, and polarization diagnostics are now complete, and the apparatus is ready to be moved onto the experimental floor. Preparations for the phase-space measurements are near completion. Once x-ray beam is available, the diagnostics experiments can begin.

The authors would like to aknowledge many useful discussions in the development of the beam diagnostics with I. McNulty. *Z. Cai, P. Ilinski, W. Yun, B. Lai, D. Legnini, and E. Gluskin*

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High-Resolution Monochromators for Nuclear Resonant Scattering

Nuclear resonant scattering experiments with synchrotron radiation (SR) aim at exciting extremely narrow energy resonances, on the order of 10^{-9} eV wide, with an initially wide bandwidth x-ray beam. Because the x-ray beam from an undulator source and standard silicon monochromator typically has an energy bandwidth on the order of 1 eV, the ratio of the nuclear resonant scattering (signal) to nonresonant scattering (background) is typically 10^{-9}. Overcoming this incredibly small signal-to-background ratio can be accomplished with a combination of techniques.

One technique involves using the time delay that exists between nuclear resonant scattering events and nonresonant scattering events, which are prompt with the incident synchrotron burst, to filter the resonant events from the background. However, this alone is not sufficient due to the difficulty in performing single photon discrimination amidst very high counting rates (10^{10} - 10^{13} photons/s). Although significant progress has been made in this area with the advent of avalanche photo-diode detectors[1]; more signal-to-background enhancement is necessary. Considerable enhancement of the signal-to-background ratio can be gained via additional monochromatization of a premonochromated x-ray beam. This motivates a need for a high-energy-resolution monochromator, or “supermono”, suitable for hard x-rays. In the x-ray energy range of 5-30 keV, "high resolution" means an energy resolution on the order of milli-eV. The APS beamline (ID3) dedicated to nuclear resonant scattering experiments will have an undulator insertion device, therefore the supermono must be designed to accept the divergence of this source.

Monochromating SR in the 5-30 keV x-ray region to the milli-eV level poses two primary problems. One involves accepting the full transverse phase space of the beam. This translates into accepting as much of the angular divergence and beam size as possible. Another problem is energy alignment. There are many practical difficulties that must be overcome in order to control the energy alignment of the monochromator. These include angular control of the crystals in terms of resolution, stability, and reproducibility. The temperature stability also becomes an increasing concern as one approaches higher energy resolution.

We have constructed and tested a design that meets the requirements of large angular acceptance, ~20+ µrad, and few milli-eV energy resolution. Further, we have produced different monochromators based on the same principle for energies ranging from 8.4 keV to 23.9 keV. The concept was initially proposed by Ishikawa, et al.,[2] and a working design was first proven by the SRI CAT nuclear resonant scattering group.[3,4] The design involves two crystals placed in a (+m, +n) Bragg geometry. The first crystal is asymmetrically cut and uses a low-order reflection to increase the angular acceptance as well as to collimate the incident synchrotron beam. The collimated beam then diffracts from a high-order reflection of a second crystal that may also be asymmetrically cut. The cut of the second crystal, if asymmetrical, is in the opposite sense as compared to the first crystal and further reduces the energy bandwidth. The two crystals produce the required milli-eV energy bandwidth with an angular acceptance that is comparable to the divergence from an undulator source. Because our needs require the monochromator to preserve the direction and size of the beam, the two crystal reflections are actually the faces of two separate channel-cut crystals. The resulting (+m, +n, -n, -m) scattering geometry performs this function and is shown in Figure 1.

Controlling the energy alignment of supermono requires controlling the angular positions of the crystals. This is achieved by mounting the crystals on rotation stages driven with piezo-electric inchworms (Burleigh RS-75). To monitor the angular positions of the crystals adequately requires angular encoders with very high precision. The necessary angular precision must be at least as good as the difference between the angular acceptance of the second crystal and the beam divergence after the first crystal. This becomes a limitation as one attempts to improve the energy resolution. For example, a 6-meV supermono that we constructed requires an angular stability of ≈0.15 arcseconds in order to maintain energy alignment within 6 meV, and a 3 meV supermono we built for the same x-ray energy requires an angular stability of ≈0.07 arcseconds. The angle encoders we used, Heidenhain ROD-800's with 1024-point interpolators, are adequate

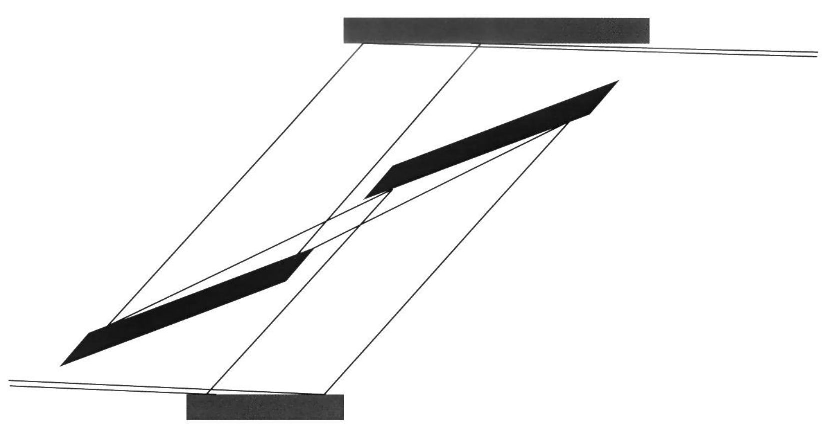

Fig. 1 The nested design of a high-energy-resolution monochromator, or supermono, for 14.4-keV x-rays. The first and fourth reflections, silicon (4 4 0), are part of a channel-cut crystal, which has an asymmetry cut of 24.7°. The second and third reflections, silicon (9 7 5), are also part of a channel-cut crystal, which has an asymmetry cut of 71.0°.

for both of these monochromators. Together they provide a resolution of 0.036 arcseconds.

The extremely narrow energy bandwidths associated with nuclear resonant scattering allow measurement of both the energy resolution and energy stability of the monochromators. The most recent supermono we have constructed monochromates SR at 14.4 keV (corresponding to the lowest nuclear resonance of ^{57}Fe) to an energy bandwidth of 3.0 meV (FWHM). This degree of monochromatization corresponds to a $\Delta E/E \approx 2\times10^{-7}$. The measured energy resolution is shown in Figure 2, in which we plot the intensity of the nuclear resonant fluorescence as a function of the relative energy alignment of the monochromator. In addition, the supermono has an angular acceptance of 22 µrad, which is reasonably well matched to the expected divergence of a typical undulator source at the APS. Also noteworthy is the fact that the design allows tuning over a wide energy range. Typically, the energy range may be 50 eV-1000 eV, depending on the actual configuration. Because energy tuning is performed by a simple rotation of the crystals, energy may be changed quite rapidly; tens of eV can be scanned in a matter of seconds. Moreover, a higher energy resolution usually results in a smaller tuning range.

In addition to reducing the amount of nonresonant scattering in nuclear resonant scattering experiments, a supermono can be used for milli-eV resolved inelastic scattering measurements. We used a supermono consisting of asymmetrically cut silicon (4 2 2) and symmetrically cut silicon (10 6 4), which has an energy resolution of ≈6 meV, to perform inelastic nuclear resonant scattering measurements on samples containing ^{57}Fe. Using the undulator source on the NE#3 beamline of the Tristan accumulator ring at KEK in Tsukuba, Japan, we were able to measure vibrational excitations in a $SrFeO_X$ sample. The SrFeOx sample undergoes a structural phase transition as the oxygen content (x) is varied from 2.5 to 3.0. The change from one local coordination (octahedral in this case) to another (tetragonal) results in different phonon spectra for the two samples. Using the supermono to scan the incident beam energy while detecting the nuclear resonant fluorescence from the ^{57}Fe within the SrFeOx sample results in the inelastic excitation spectrum of Figure 3. Thus, we were able to measure an inelastic excitation spectrum using hard x-rays (14.4 keV) with 6-meV energy resolution. From this inelastic excitation spectrum, it is possible to recover the phonon density of states for the Fe sites[5].

We have designed and tested high-energy-resolution monochromators, or supermonos, for x-rays in the energy range of 5-30 keV, that possess energy resolutions as low as 3 meV. These monochromators will accept a large portion of the phase space produced by an undulator source at the APS. Supermonos have applications in both nuclear resonant scattering experiments and milli-eV-resolved inelastic scattering measurements. In the future, we hope to achieve energy resolutions better than 3 meV in this energy range.

We would like to acknowledge X. Zhang and Y. Yoda for their support during the testing of the 3 meV supermono and during the milli-eV inelastic scattering measurements at

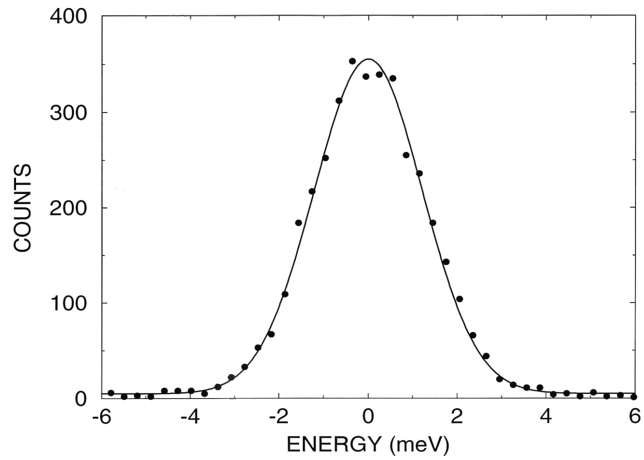

Fig. 2 Energy scan of the (4 4 0)-(9 7 5) supermono of Figure 1 using the nuclear fluorescence of the 14.413-keV level in ^{57}Fe. The FWHM is 3.0 meV. This degree of monochromatization corresponds to a $\Delta E/E \approx 2 \times 10^{-7}$.

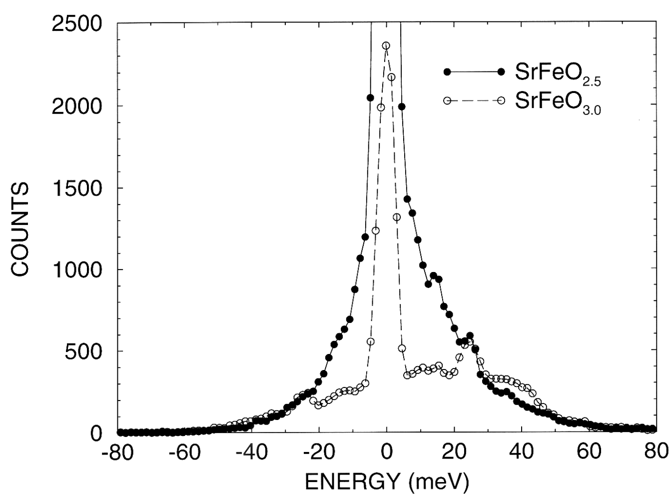

Fig. 3 Inelastic nuclear resonant scattering spectra taken with a (4 2 2)-(10 6 4) supermono, which has an energy resolution of 6 meV. The sample is $SrFeO_X$ with x=2.5 and x=3.0 (95% enriched in ^{57}Fe). The different crystal structures associated with the different oxygen contents are responsible for the altered inelastic spectra.

KEK. We would also like to acknowledge the support of the staff of CHESS at Cornell University where much of the testing of supermonos was carried out. *T.S. Toellner, T.M. Mooney, W. Sturhahn, and E.E. Alp.*

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Calendar

April 14
Began moving to our lab office module

June 29
Research Directorate Meeting

WHO'S NEW

Peter Mui Postdoctoral Research Associate

Peter Mui is working with George Srajer on the *in-situ* monitoring system for synchrotron optics in a high-heat-load environment program. Peter got his Ph.D. in electrical engineering from the University of Arizona. He also holds a masters degree in mathematics.

SRI CAT
Synchrotron Radiation Instrumentation
Collaborative Access Team
Argonne National Laboratory
9700 South Cass Avenue
Argonne Il 60439

Executive Director
Dennis M. Mills

Program Director
Efim Gluskin

Associate Director
Deming Shu

SRI CAT Secretary
Cheryl Zidel
Phone: 708-252-7366
Fax: 708-252-3222
e-mail: Zidel@aps.anl.gov

Newsletter Coeditors
Albert T. Macrander
Ian McNulty

Address additions, changes and deletions are welcome. Forward them to the SRI CAT Secretary.

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Publications

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